APPENDIX C
EPA REGION 9 DATA QUALITY INDICATOR TABLES

U.S. Environmental Protection Agency

Region 9: Quality Assurance

Serving Arizona, California, Hawaii, Nevada, the Pacific Islands, and over 140 Tribal Nations

Data Quality Indicator Tables

The following tables list the EPA Region 9 quality control acceptance criteria for selected laboratory analytical methods. Region 9 has developed these specifications to ensure that laboratory-generated analytical data is of acceptable quality for use in environmental decision making. For more information on the Region 9 data review procedures, please see the Data Validation page.

The following Data Quality Indicator Tables are available in PDF format. Revisions (indicated in bold) and WordPerfect versions of these tables may be obtained by contacting the Region 9 QA program.

HARDNESS, TOTAL (mg/L as CaCO3)
EPA Method 130.2 (Titrimetric, EDTA)

Table 1. Summary of Contract Required Detection Limits, Holding Times, and Preservation for Hardness

Analytical Parameter	Contract Required Detection Limit (CRDL)	Technical and Contract Holding Times	Preservation
Hardness, Total (mg/L as $CaCO_3$)	5.0 mg/L	Technical: 6 months from collection; Contract: 6 months from receipt at laboratory	HNO_3 to pH <2; Cool to 4EC ±2EC

Follow the procedure outlined in EPA method 130.2 for the analysis of samples for hardness.
Use sample aliquots containing not more than 25 mg CaCO3 to avoid large titration volumes. This is determined by performing practice runs.
pretreat waste waters and highly polluted waters by digesting the sample as described in the procedure in Section 7.1.2 of EPA Method 130.2.

Data Calculations and Reporting Units:

Calculate the sample results according to Section 8 of EPA Method 130.2. Sample results are to be reported in the concentration unit of milligram per liter (mg/L) of CaCO3. The concentration result shall be reported to two significant figures if the result is less than 10 mg/L; and to three significant figures if the result is greater than or equal to 10 mg/L.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 2. Summary of Internal Quality Control Procedures for Hardness by EPA 130.2

QC Element	Frequency	Acceptance Criteria	Corrective Action
Titration Blank (MB)	One per Batch or SDG [a] (1 per 20 samples minimum)	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all associated samples must be prepared again with another method blank and reanalyzed
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"
One set of QC reference samples (two concentration levels)	One per batch or SDG (1 per 20 samples minimum)	± 15% of expected result	1. Terminate analysis 2. Identify and document the problem 3. Reanalyze all associated samples

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

Check the normality of titrant each day.

Add inhibitors if the end point is not sharp. Excessive amounts of heavy metals interfere by causing fading or indistinct end points. To correct for interferences, refer to Section 7.4 of EPA Method 130.2.

METHOD #: 150.1	Approved for NPDES (Editorial Revision 1978, 1982)
TITLE:	pH (Electrometric)
ANALYTE:	pH
INSTRUMENTATION:	pH Meter
STORET No.	Determined on site 00400 Laboratory 00403

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition).

2.0 Summary of Method

2.1 The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

3.0 Sample Handling and Preservation

3.1 Samples should be analyzed as soon as possible preferably in the field at the time of sampling.

3.2 High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

4.0 Interferences

4.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

4.2 Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1 + 9) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent and cannot be controlled, it should therefore be noted by reporting both the pH and temperature at the time of analysis.

5.0 Apparatus

5.1 pH Meter-laboratory or field model. A wide variety of instruments are commercially available with various specifications and optional equipment.

5.2 Glass electrode.

5.3 Reference electrode-a calomel, silver-silver chloride or other reference electrode of constant potential may be used.

NOTE 1: Combination electrodes incorporating both measuring and reference functions are convenient to use and are available with solid, gel type filling materials that require minimal maintenance.

5.4 Magnetic stirrer and Teflon-coated stirring bar.

5.5 Thermometer or temperature sensor for automatic compensation.

6.0 Reagents

6.1 Primary standard buffer salts are available from the National Bureau of Standards and should be used in situations where extreme accuracy is necessary.

6.1.1 Preparation of reference solutions from these salts require some special precautions and handling[1] such as low conductivity dilution water, drying ovens, and carbon dioxide free purge gas. These solutions should be replaced at least once each month.

6.2 Secondary standard buffers may be prepared from NBS salts or purchase as a solution from commercial vendors. Use of these commercially available solutions, that have been validated by comparison to NBS standards, are recommended for routine use.

7.0 Calibration

7.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

7.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart.

7.2.1 Various instrument designs may involve use of a "balance" or "standardize" dial and/or a slope adjustment as outlined in the manufacturer's instructions. Repeat adjustments on successive portions of the two buffer solutions as outlined in procedure 8.2 until readings are within 0.05 pH units of the buffer solution value.

8.0 Procedure

8.1 Standardize the meter and electrode system as outlined in Section 7.

8.2 Place the sample or buffer solution in a clean glass beaker using a sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar.

8.2.1 If field measurements are being made the electrodes may be immersed

directly in the sample stream to an adequate depth and moved in a manner to insure sufficient sample movement across the electrode sensing element as indicated by drift free (< 0.1 pH) readings.

8.3 If the sample temperature differs by more than 2°C from the buffer solution the measured pH values must be corrected. Instruments are equipped with automatic or manual compensators that electronically adjust for temperature differences. Refer to manufacturer's instructions.

8.4 After rinsing and gently wiping the electrodes, if necessary, immerse them into the sample beaker ar sample stream and stir at a constant rate to provide homogeneity and suspension of solids. Rate of stirring should minimize the air transfer rate at the air water interface of the sample. Note and record sample pH and temperature. Repeat measurement on successive volumes of sample until values differ by less than 0.1 pH units. Two or three volume changes are usually sufficient.

8.5 For acid rain samples it is most important that the magnetic stirrer is not used. Instead, swirl the sample gently for a few seconds after the introduction of the electrode(s). Allow the electrode(s) the equilibrate. The air-water interface should not be disturbed while measurement is being made. If the sample is not in equilibrium with the atmosphere, pH values will change as the dissolved gases are either absorbed or desorbed. Record sample pH and temperature.

9.0 Calculation

9.1 pH meters read directly in pH units. Report pH to the nearest 0.1 unit and temperature to the nearest degree °C.

10.0 Precision and Accuracy

10.1 Forty-four analysts in twenty laboratories analyzed six synthetic water samples containing exact increments of hydrogen-hydroxyl ions, with the following results:

pH Units	Standard Deviation pH Units	Accuracy as Bias, %	Bias, pH Units
3.5	0.10	-0.29	-0.01
3.5	0.11	-0.00	
7.1	0.20	+1.01	+0.07
7.2	0.18	-0.03	-0.002
8.0	0.13	-0.12	-0.01
8.0	0.12	+0.16	+0.01

(FWPCA Method Study 1, Mineral and Physical Analyses)

10.2 In a single laboratory (EMSL), using surface water samples at an average pH of 7.7, the standard deviation was± 0.1.

TOTAL DISSOLVED SOLIDS (TDS)
EPA Method 160.1 (Gravimetric, Dried at 180EC)

Table 1. Summary of Contract Required Detection Limits, Holding Times, and Preservation for Total Dissolved Solids (TDS)

Analytical Parameter	Contract Required Detection Limit (CRDL)	Technical and Contract Holding Times	Preservation
Total Dissolved Solids (TDS)	20 mg/L	Technical: 7 days from collection; Contract: 5 days from receipt at laboratory	Cool to 4EC ±2EC

Follow the procedure outlined in EPA method 160.1 for the analysis of samples for TDS.

Weigh solid residue to a constant weight, defined as two consecutive weight measurements differing by less than 0.5 mg, or less than 4%, whichever is smaller.

Data Calculations and Reporting Units:

Calculate the sample results according to Section 8 of EPA Method 160.1.

Report sample results in concentration units of milligram per liter (mg/L) as total dissolved solids. Report TDS concentrations that are less than 100 mg/L to 2 significant figures, and TDS concentrations that are greater than or equal to 100 mg/L to 3 significant figures.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 2. Summary of Internal Quality Control Procedures for Total Dissolved Solids (TDS) by EPA 160.1

QC Element	Frequency	Acceptance Criteria	Corrective Action
Analytical Balance Check: Weights of 100 mg, 1 g, and 100 g	Daily	Difference < 0.5 mg	1. Identify and document problem 2. Verify before sample analysis
Method Blank (MB)	One per Batch or SDG[a] (1 per 20 samples minimum)	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all associated samples must be prepared again with another method blank and reanalyzed
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"
One set (two concentration levels) mineral reference samples	One set per batch or SDG (1 set per 20 samples minimum)	± 15% from expected concentration	1. Terminate analysis 2. Identify, document, and correct the problem 3. Reanalyze all associated samples

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

Use sample aliquots of 100 mL. If the residue in a sample is greater than 200 mg, repeat the analysis using

a smaller sample aliquot.

METHOD #: 160.2	Approved for NPDES (Issued 1971)
TITLE:	Residue, Non-Filterable (Gravimetric, Dried at 103-105°C)
ANALYTE:	Residue ,Non-Filterable
INSTRUMENTATION:	Drying Oven
STORET No.	00530

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
1.2 The practical range of the determination is 4 mg/L to 20,000 mg/L.

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C.
2.2 The filtrate from this method may be used for Residue, Filterable.

3.0 Definitions

3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103-105°C.

4.0 Sample Handling and Preservation

4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.

6.0 Apparatus

6.1 Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeves Angel 934-AH, Gelman type A/E, or equivalent.
NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.

6.2 Filter support: filtering apparatus with reservoir and a coarse (40-60 microns) fritted disc as a filter support.
NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

6.3 Suction flask.

6.4 Drying oven, 103-105°C.

6.5 Desiccator.

6.6 Analytical balance, capable of weighing to 0.1 mg.

7.0 Procedure

7.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

7.2 Selection of Sample Volume
For a 4.7 cm diameter filter, filter 100 mL of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue.If other filter diameters are used, start with a sample volume equal to 7 mL/cm^2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.
NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five mL increments for timing are suggested. Continue to record the time and volume increments until filtration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration rate occurred.

7.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

7.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove

all traces of water by continuing to apply vacuum after sample has passed through.

7.5 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.
NOTE: Total volume of wash water used should equal approximately 2 mL per cm^2. For a 4.7 cm filter the total volume is 30 mL.

7.6 Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103-105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

8.0 Calculations

8.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/L} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = mL of sample filtered

9.0 Precision and Accuracy

9.1 Precision data are not available at this time.
9.2 Accuracy data on actual samples cannot be obtained.

Bibliography

1. NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.

METHOD 180.1

DETERMINATION OF TURBIDITY BY NEPHELOMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

METHOD 180.1

DETERMINATION OF TURBIDITY BY NEPHELOMETRY

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of turbidity in drinking, ground, surface, and saline waters, domestic and industrial wastes.

1.2 The applicable range is 0-40 nephelometric turbidity units (NTU). Higher values may be obtained with dilution of the sample.

2.0 SUMMARY OF METHOD

2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTU's, are made in a nephelometer designed according to specifications given in Sections 6.1 and 6.2. A primary standard suspension is used to calibrate the instrument. A secondary standard suspension is used as a daily calibration check and is monitored periodically for deterioration using one of the primary standards.

2.1.1 Formazin polymer is used as a primary turbidity suspension for water because it is more reproducible than other types of standards previously used for turbidity analysis.

2.1.2 A commercially available polymer primary standard is also approved for use for the National Interim Primary Drinking Water Regulations. This standard is identified as AMCO-AEPA-1, available from Advanced Polymer Systems.

3.0 DEFINITIONS

3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogates analytes.

3.2 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.3 **Laboratory Reagent Blank (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method

analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.4 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear.

3.5 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.6 **Primary Calibration Standard (PCAL)** -- A suspension prepared from the primary dilution stock standard suspension. The PCAL suspensions are used to calibrate the instrument response with respect to analyte concentration.

3.7 **Quality Control Sample (QCS)** -- A solution of the method analyte of known concentrations that is used to fortify an aliquot of LRB matrix. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance.

3.8 **Secondary Calibration Standards (SCAL)** -- Commercially prepared, stabilized sealed liquid or gel turbidity standards calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymers.

3.9 **Stock Standard Suspension (SSS)** -- A concentrated suspension containing the analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Stock standard suspension is used to prepare calibration suspensions and other needed suspensions.

4.0 INTERFERENCES

4.1 The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.

4.2 The presence of true color, that is the color of water which is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking waters.

4.3 Light absorbing materials such as activated carbon in significant concentrations can cause low readings.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in

this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 Hydrazine Sulfate (Section 7.2.1) is a carcinogen. It is highly toxic and may be fatal if inhaled, swallowed, or absorbed through the skin. Formazin can contain residual hydrazine sulfate. Proper protection should be employed.

6.0 EQUIPMENT AND SUPPLIES

6.1 The turbidimeter shall consist of a nephelometer, with light source for illuminating the sample, and one or more photo-electric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter should be designed so that little stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period.

6.2 Differences in physical design of turbidimeters will cause differences in measured values for turbidity, even though the same suspension is used for calibration. To minimize such differences, the following design criteria should be observed:

6.2.1 Light source: Tungsten lamp operated at a color temperature between 2200-3000°K.

6.2.2 Distance traversed by incident light and scattered light within the sample tube: Total not to exceed 10 cm.

6.2.3 Detector: Centered at 90° to the incident light path and not to exceed ±30° from 90°. The detector, and filter system if used, shall have a spectral peak response between 400 nm and 600 nm.

6.3 The sensitivity of the instrument should permit detection of a turbidity difference of 0.02 NTU or less in waters having turbidities less than 1 unit. The instrument should measure from 0-40 units turbidity. Several ranges may be necessary to obtain both adequate coverage and sufficient sensitivity for low turbidities.

6.4 The sample tubes to be used with the available instrument must be of clear, colorless glass or plastic. They should be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched. A light coating of silicon oil may be used to mask minor imperfections in glass tubes. They must not be handled at all where the light strikes them, but should be provided with sufficient extra length, or with a protective case, so that they may be handled. Tubes should be checked, indexed and read at the orientation that produces the lowest background blank value.

6.5 Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.6 Glassware -- Class A volumetric flasks and pipets as required.

7.0 REAGENTS AND STANDARDS

7.1 Reagent water, turbidity-free: Pass deionized distilled water through a 0.45μ pore size membrane filter, if such filtered water shows a lower turbidity than unfiltered distilled water.

7.2 Stock standard suspension (Formazin):

7.2.1 Dissolve 1.00 g hydrazine sulfate, $(NH_2)_2.H_2SO_4$ (CASRN 10034-93-2) in reagent water and dilute to 100 mL in a volumetric flask. **CAUTION**--carcinogen.

7.2.2 Dissolve 10.00 g hexamethylenetetramine (CASRN 100-97-0) in reagent water and dilute to 100 mL in a volumetric flask. In a 100 mL volumetric flask, mix 5.0 mL of each solution (Sections 7.2.1 and 7.2.2). Allow to stand 24 hours at 25 ±3°C, then dilute to the mark with reagent water.

7.3 Primary calibration standards: Mix and dilute 10.00 mL of stock standard suspension (Section 7.2) to 100 mL with reagent water. The turbidity of this suspension is defined as 40 NTU. For other values, mix and dilute portions of this suspension as required.

7.3.1 A new stock standard suspension (Section 7.2) should be prepared each month. Primary calibration standards (Section 7.3) should be prepared daily by dilution of the stock standard suspension.

7.4 Formazin in commercially prepared primary concentrated stock standard suspension (SSS) may be diluted and used as required. Dilute turbidity standards should be prepared daily.

7.5 AMCO-AEPA-1 Styrene Divinylbenzene polymer primary standards are available for specific instruments and require no preparation or dilution prior to use.

7.6 Secondary standards may be acceptable as a daily calibration check, but must be monitored on a routine basis for deterioration and replaced as required.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with turbidity free water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2 No chemical preservation is required. Cool sample to 4°C.

8.3 Samples should be analyzed as soon as possible after collection. If storage is required, samples maintained at 4°C may be held for up to 48 hours.

9.0 QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and analysis of laboratory reagent blanks and other solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE.

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS).

9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ±10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analysis of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analyses.

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment.

9.3.2 Instrument Performance Check Solution (IPC) -- For all determinations, the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every tenth sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is

within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data. NOTE: Secondary calibration standards (SS) may also be used as the IPC.

9.3.3 Where additional reference materials such as Performance Evaluation samples are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Turbidimeter calibration: The manufacturer's operating instructions should be followed. Measure standards on the turbidimeter covering the range of interest. If the instrument is already calibrated in standard turbidity units, this procedure will check the accuracy of the calibration scales. At least one standard should be run in each instrument range to be used. Some instruments permit adjustments of sensitivity so that scale values will correspond to turbidities. Solid standards, such as those made of lucite blocks, should never be used due to potential calibration changes caused by surface scratches. If a pre-calibrated scale is not supplied, calibration curves should be prepared for each range of the instrument.

11.0 PROCEDURE

11.1 Turbidities less than 40 units: If possible, allow samples to come to room temperature before analysis. Mix the sample to thoroughly disperse the solids. Wait until air bubbles disappear then pour the sample into the turbidimeter tube. Read the turbidity directly from the instrument scale or from the appropriate calibration curve.

11.2 Turbidities exceeding 40 units: Dilute the sample with one or more volumes of turbidity-free water until the turbidity falls below 40 units. The turbidity of the original sample is then computed from the turbidity of the diluted sample and the dilution factor. For example, if 5 volumes of turbidity-free water were added to 1 volume of sample, and the diluted sample showed a turbidity of 30 units, then the turbidity of the original sample was 180 units.

11.2.1 Some turbidimeters are equipped with several separate scales. The higher scales are to be used only as indicators of required dilution volumes to reduce readings to less than 40 NTU.

Note: Comparative work performed in the Environmental Monitoring Systems Laboratory - Cincinnati (EMSL-Cincinnati) indicates a progressive error on sample turbidities in excess of 40 units.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Multiply sample readings by appropriate dilution to obtain final reading.

12.2 Report results as follows:

NTU	Record to Nearest:
0.0 - 1.0	0.05
1 - 10	0.1
10 - 40	1
40 - 100	5
100 - 400	10
400 - 1000	50
>1000	100

13.0 METHOD PERFORMANCE

13.1 In a single laboratory (EMSL-Cincinnati), using surface water samples at levels of 26, 41, 75, and 180 NTU, the standard deviations were ±0.60, ±0.94, ±1.2, and ±4.7 units, respectively.

13.2 The interlaboratory precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in NTU.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American

Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202)872-4477.

15.0 WASTE MANAGEMENT

15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3.

16.0 REFERENCES

1. Annual Book of ASTM Standards, Volume 11.01 Water (1), Standard D1889-88A, p. 359, (1993).

2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, pp. 2-9, Method 2130B, (1992).

Metals by Inductively Coupled Plasma (ICP) Atomic Emission Spectroscopy (AES)
EPA Method 200.7

Table 1A. Summary of Holding Times and Preservation for Metals

Analytical Parameter [a]	Technical and Contract Holding Times	Preservation
Metals in water	Technical: 180 days from date of collection; Contract: 35 days from sample receipt at laboratory	pH <2 (with nitric acid)
Metals in soil	Technical: 180 days from date of collection; Contract: 35 days from sample receipt at laboratory	Cool to 4EC ±2EC

[a] Individual target metals are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the sample results according to the protocol of the appropriate instrument data system.

Report water sample results in concentration units of micrograms per liter (Fg/L), and soil sample results in concentration units of milligrams per kilogram (mg/kg) on a dry weight basis. Report percent solids to the nearest percent.

For rounding results, adhere to the following rules:
a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Analyte List, CAS Numbers, and Contract Required Detection Limits for Metals by ICP-AES

ANALYTE	CAS No.	CRDL for Water (µg/L)	CRDL for Soil (mg/Kg)
Aluminum (Al)	7429-90-5	200	40
Antimony (Sb)	7440-36-0	60	12
Arsenic (As)	7440-38-2	10	2
Barium (Ba)	7440-39-3	200	40
Beryllium (Be)	7440-41-7	5	1
Boron (B)	**7440-42-8**	**10**	**2**
Cadmium (Cd)	7440-43-9	5	1
Calcium (Ca)	7440-70-2	5000	1000
Chromium (Cr)	7440-47-3	10	2
Cobalt (Co)	7440-48-4	50	10
Copper (Cu)	7440-50-8	25	5
Iron (Fe)	7439-89-6	100	20
Lead (Pb)	7439-92-1	3	1
Magnesium (Mg)	7439-95-4	5000	1000
Manganese (Mn)	7439-96-5	15	3
Molybdenum (Mo)	7439-97-6	20	4
Nickel (Ni)	7440-02-0	40	8
Potassium (K)	7723-14-0	5000	1000
Selenium (Se)	7782-49-2	5	1
Silica (SiO2)	**7631-86-9**	**100**	**20**
Silver (Ag)	7440-22-4	10	2
Sodium (Na)	7440-23-5	5000	1000
Thallium (Tl)	7440-28-0	10	2
Vanadium (V)	7440-62-2	20	4
Zinc (Zn)	7440-66-6	10	2

Table 2A. Summary of Calibration Procedures for Metals by ICP-AES

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 1 calibration standard) (ICAL)	Initially, Daily; whenever required, due to failure of CCV	Acceptable ICV, CRI, and ICB standards	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	90-110% of expected concentration	1. Terminate analysis and identify and document problem 2. Reprep and re-analyze ICV and all associated samples 3. Re-calibrate and re-analyze reprepped ICV and all associated samples
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine Source of contamination 3. Reprep ICB and CCB 4. Re-analyze all samples associated with a contaminated blank
Continuing Calibration Verification (CCV)	Before samples, after every 10 samples, and end of run	90-110% of expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last acceptable CCV
Contract Required Detection Limit Verification Standard (CRI)[a]	After ICV, but before sample analysis	65-135% of expected concentration	1. Re-prep and re-analyze standard 2. Re-calibrate and verify
ICP Interference Check Samples (ICS)	Run at start and finish of daily run or twice per 8 hours	80-120% of expected concentration	1. Re-prep and re-analyze standard 2. Re-calibrate, verify, and re-analyze all associated samples

[a] The CRI standard must be between the CRDL and 2X CRDL.

Table 3. Summary of Internal Quality Control Procedures for Metals by ICP-AES

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per batch or SDG [a, b]	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all affected samples must be prepared again with another method blank and re-analyzed
Duplicate Sample (DUP)	One per batch or SDG [a, b]	**Waters:** RPD <± 20 for samples >5X CRDL; ± CRDL for samples <5X CRDL **Soils:** RPD <± 35 for samples >5X CRDL; ± 2xCRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike Sample (MS)	One per batch or SDG [a, b]	± 75-125% of expected value [c]	1. Flag associated data with an "N"
Laboratory Control Sample (LCS)	One per batch or SDG [a, b]	**Waters:** 80-120% of expected concentration **Soils:** within control limits of certified solid LCS or 80-120% of expected concentration	1. Terminate analysis and identify and document the problem 2. Re-analyze all associated samples
Serial Dilution Sample (5 X Dilution)	One per batch or SDG [a, b]	± 10% difference from original results for analyte concentrations greater than 50 times the IDL	1. Flag associated data with a "E"

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within case; or each 7 calendar day period during which field samples in a case are received.
[b] Minimum requirement is the analysis of 1 QC sample per 20 samples.
[c] An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of 4. In such an event, the data shall be reported unflagged.

Dilute and re-analyze samples with concentrations exceeding the linear range. Results for such re-analyses should fall within the mid-range of the linear range. Report results and submit documentation for both analyses.

DETERMINATION OF TRACE ELEMENTS IN WATERS AND WASTES
BY INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY
EPA Method 200.8 (Revision 5.4, 1994)

Table 1A. Summary of Holding Times and Preservation for Trace Elements by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Analytical Parameter [a]	Technical and Contract Holding Times	Preservation
Trace Elements in Water	Technical without mercury: 180 days from collection; Technical with mercury: 28 days from collection; Contract without mercury: 35 days from receipt at laboratory; Contract with mercury: 26 days from receipt at laboratory;	HNO_3 to pH <2; Cool to 4EC ±2EC
Trace Elements in Soil	Technical without mercury: 180 days from collection; Technical with mercury: 28 days from collection; Contract without mercury: 35 days from receipt at laboratory; Contract with mercury: 26 days from receipt at laboratory;	Cool to 4EC ±2EC

[a] Individual target elements are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the concentration of individual elements according to the equation specified in Section 12.0 of Method 200.8. Report water sample results in concentration units of micrograms per liter (Fg/L).

Report soil sample results on a dry-weight basis in milligrams per kilogram (mg/kg). Report percent solid and percent moisture to the nearest whole percentage point.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Elements List, CAS Numbers, and Contract Required Detection Limits (CRDL) for Trace Elements by ICP-MS

COMPOUND	CAS No.	CRDL for Water (µg/L)	CRDL for Soil (mg/Kg)
Aluminum	7429-90-5	30	15
Antimony	7440-36-0	2.0	1.0
Arsenic	7440-38-2	1.0	0.5
Barium	7440-39-3	10	5.0
Beryllium	7440-41-7	1.0	0.5
Cadmium	7440-43-9	1.0	0.5
Chromium	7440-47-3	2.0	1.0
Cobalt	7440-48-4	0.5	0.25
Copper	7440-50-8	2.0	1.0
Lead	7439-92-1	1.0	0.5
Manganese	7439-96-5	0.5	0.25
Mercury	7439-97-6	0.2	0.1
Molybdenum	**7439-98-7**	**1.0**	**0.5**
Nickel	7440-02-0	1.0	0.5
Selenium	7782-49-2	5.0	2.5
Silver	7440-22-4	1.0	0.5
Thallium	7440-28-0	1.0	0.5
Thorium	**7440-29-1**	**1.0**	**0.5**
Uranium	**7440-61-1**	**1.0**	**0.5**
Vanadium	7440-62-2	1.0	0.5
Zinc	7440-66-6	1.0	0.5

Table 2. Summary of Calibration Procedures for Trace Elements by EPA Method 200.8

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
ICP-MS Precalibration Routine [a]	Daily prior to instrument calibration	Criteria specified in Section 10.2 of Method 200.8	1. Identify the problem. 2. Tuning criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 1 calibration standard) (ICAL)	Initially, Daily; whenever required, due to failure of IPC, QCS, or CRDL	Acceptable IPC, QCS, and CRDL standards	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Instrument Performance Check (IPC) (mid range calibration standard)	Following the calibration and prior to sample analysis; after every 10 samples; and end of run	90-110% of expected value	1. Recalibrate and verify 2. Reanalyze samples back to last good QCS
Quality Control Sample (QCS) (Separate source from ICAL standards)	Following the IPC and prior to sample analysis	90-110% of expected value or limits listed in Table 8 of Method 200.8, whichever is greater	1. Recalibrate and verify 2. Reanalyze samples back to last compliant IPC
Calibration Blank Verification	After ICAL; every IPC; and end of the analytical sequence	< CRDL	1. Terminate analysis 2. Identify and document the problem 3. Recalibrate, verify and reanalyze all associated samples
Contract Required Detection Limit (CRDL) Verification Standard	After QCS, but before sample analysis	65-135% of expected concentration	1. Reprep and reanalyze standard 2. Recalibrate and verify
Internal Standards (IS) [b]	In all samples, standards, and blanks	IS area within 60-125% of the IS area in the calibration blank	1. Reanalyze all samples analyzed while system was out-of-control

[a] Perform precalibration routine specified in Section 10.2 of Method 200.8. Instrument stability must be demonstrated by running the tuning solution a minimum of five times with resulting relative standard deviations (RSD) of absolute signals for all analytes of less than 5%.

[b] Section 10.3 of Method 200.8 identifies Table 3 for a list of acceptable internal standards. A minimum of three internal standards must be used.

Table 3. Summary of Internal Quality Control Procedures for Trace Elements by EPA Method 200.8

QC Element	Frequency	Acceptance Criteria	Corrective Action
Laboratory Reagent Blank (LRB)	One per Batch or SDG [a] (1 per 20 samples minimum)	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all associated samples must be prepared again with another method blank and reanalyzed
Laboratory Fortified Blank (LFB)	One per batch or SDG (1 per 20 samples minimum)	85-115% of expected value	1. Terminate analysis 2. Identify and document the problem 3. Reanalyze all associated samples
Laboratory Fortified Matrix (LFM) [b]	A minimum of 10% of the field samples	70-130% of expected value	1. Flag associated data with an "N"
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each seven (7) calendar day period during which field samples in a case are received.

[b] If the LFM sample exceeds the calibration range, the sample must be diluted appropriately, re-spiked, and reanalyzed. LFM recovery calculations are not required if the concentration of the analyte added is <30%.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

METHOD #: 212.3	Approved for NPDES (Issued 1974)
TITLE:	Boron (Colorimetric, Curcumin)
ANALYTE:	CAS # B Boron 7440-42-8
INSTRUMENTATION:	Spectrophotometer
STORET No.	Total 01022 Dissolved 01020 Suspended 01021

1.0 Scope and Application

1.1 This colorimetric method finds maximum utility for waters whose boron content is below 1 mg/L.

1.2 The optimum range of the method on undiluted or unconcentrated samples is 0.1-1.0 mg/L of boron.

1.3 This method is applicable to drinking, and surface waters, domestic and industrial wastes.

2.0 Summary of Method

2.1 When a sample of water containing boron is acidified and evaporated in the presence of curcumin, a red-colored product called rosocyanine is formed. The rosocyanine is taken up in a suitable solvent, and the red color is compared with standards photometrically.

3.0 Comments

3.1 Nitrate nitrogen concentrations above 20 mg/L interfere.

3.2 Significantly high results are possible when the total of calcium and magnesium hardness exceeds 100 mg/L as $CaC0_3$. Passing the sample through a cation exchange resin eliminates this problem.

3.3 Close control of such variables as volumes and concentrations of reagents, as well as time and temperature of drying, must be exercised for maximum accuracy.

3.4 Data to be entered into STORET must be reported as µg/L.

4.0 Precision and Accuracy

4.1 A synthetic sample prepared by the Analytical Reference Service, PHS, containing 240 µg/L B, 40 µg/L As, 250 µg/L Be, 20 µg/L Se, and 6 µg/L V in distilled water, was analyzed by the curcumin method with a relative standard deviation of 22.8% and a relative error of 0% in 30 laboratories.

5.0 Reference

5.1 The procedure to be used for this determination is found in: Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 287, Method 405A (1975).

Mercury in Water by Manual Cold Vapor Atomic Absorption (CVAA)
EPA Method 245.1

Table 1A. Summary of Holding Times and Preservation for Mercury

Analytical Parameter	Technical and Contract Holding Times	Preservation
Mercury in water	Technical: 28 days from date of collection; Contract: 26 days from sample receipt at laboratory	pH <2 (with nitric acid)

Data Calculations and Reporting Units:

Calculate the sample results by comparing sample peak height, area, or absorbance against the standard curve.

Report water sample results in concentration units of micrograms per liter (Fg/L), and soil sample results in concentration units of milligrams per kilogram (mg/kg) on a dry weight basis. Report percent solids to the nearest percent.

For rounding results, adhere to the following rules:
a) If the number following those to be retained is less than 5, round down;
b) If the number following those to be retained is greater than 5, round up; or
c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Analyte List, CAS Numbers, and Contract Required Detection Limit Mercury CVAA

ANALYTE	CAS No.	CRDL for Water (µg/L)
Mercury	7439-97-6	0.2

Table 2. Summary of Calibration Procedures for Mercury in Water by Manual CVAA

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 standards) (ICAL) [a]	Initially, each analytical batch; whenever required, due to failure of CCV	r $ 0.995	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	80-120% of expected concentration	1. Terminate analysis and identify and document problem 2. Reprep and re-analyze ICV and all associated samples 3. Re-calibrate and re-analyze reprepped ICV and all associated samples
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine Source of contamination 3. Reprep ICB and CCB 4. Re-analyze all samples associated with a contaminated blank
Continuing Calibration Verification (CCV)	Before Samples, after every 10 samples, and end of run	80-120% of expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last acceptable CCV
Contract Required Detection Limit Verification Standard (CRA)	After ICV, but before sample analysis	65-135 of expected concentration	1. Reprep and re-analyze standard 2. Re-calibrate and verify

[a] The ICAL low standard must be at the CRDL. The low standard instrument response should be calculated from the linear regression. The predicted result must be within 25% of the low standard true value.

Table 3. Summary of Internal Quality Control Procedures for Mercury in Water by Manual CVAA

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per batch or SDG [a, b]	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all affected samples must be prepared again with another method blank and re-analyzed
Duplicate Sample (DUP)	One per batch or SDG [a, b]	RPD <± 20 for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike Sample (MS)	One per batch or SDG [a, b]	75-125% of expected value [c]	1. Flag associated data with an "N"
Laboratory Control Sample (LCS)	One per batch or SDG [a, b]	80-120% of expected concentration	1. Terminate analysis and identify and document the problem 2. Re-analyze all associated samples

[a] SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 7 calendar day period during which field samples in a case are received.

[b] Minimum requirement is the analysis of 1 QC sample per 20 samples.

[c] An exception to this rule is granted in situations where the sample concentration exceeds the spike concentration by a factor of 4. In such an event, the data shall be reported unflagged.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.